AD-A093 655 NATIONAL RESEARCH COUNCIL OF CANADA OTTAWA (ONTARIO) --ETC F/G 11/8
PROPERTIES OF BASE STOCKS OBTAINED FROM USED ENGINE OILS BY ACI--ETC(U)
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PROPERTIES OF BASE STOCKS OBTAINED FROM USED ENGINE OILS BY ACID/CLAY RE-REFINING

by

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OTTAWA

SEPTEMBER 1980

Canada

MECHANICAL ENGINEERING REPORT
MP-75
NRC NO. 18719

PROPERTIES OF BASE STOCKS OBTAINED FROM
USED ENGINE OILS BY ACID/CLAY RE-REFINING

(PROPRIÉTÉS DES STOCKS DE BASE OBTENUS PAR REGÉNÉRATION DES HUILES
À MOTEUR USÉES PAR LE PROCÉDÉ DE TRAITEMENT À L'ACIDE ET À LA TERRE

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SUMMARY

For more than 10 years the Fuels and Lubricants Laboratory of the Division of Mechanical Engineering has been examining the properties of base stocks, used oil feedstocks and re-refined engine oils of Canadian re-refiners. Over 20 samples of base stocks from six Canadian re-refiners have been examined. Data are presented and compared with virgin base stock data. In addition, tables of suggested specification limits for base stocks and batch-to-batch consistency ranges are given. Finally, data are given for a number of samples obtained from a re-refiner in India.

As shown, when well re-refined, the base stocks have excellent properties including a good response to anti-oxidants and a high degree of cleanliness.

SOMMAIRE

Pour une période de 10 ans le Laboratoire des combustibles et des lubrifiants de la Division de génie mécanique a vérifié les propriétés des stocks de base, des stocks d'alimentation d'huiles usées et des huiles à moteur regénériés par les regénérateurs canadiens. Plus de vingt échantillons de stock de base provenant de six regénérateurs canadiens ont été vérifiés. Les résultats sont inscrits et comparés avec les résultats des stocks de base d'huiles pures. En plus, les tableaux de normes limites suggérées ainsi que la gamme d'uniformité de lot en lot sont inclus. Finalement, les résultats obtenus sur quelques échantillons provenant d'un regénérateur de l'Inde sont également inclus.

Tel que démontré, quand bien regénérés, les stocks de base ont des propriétés excellentes incluant une bonne réaction aux anti-oxydants ainsi qu'un haut degré de propreté.

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1.0 INTRODUCTION

Owing to limited laboratory facilities of Canadian re-refineries, the NRC Fuels and Lubricants Laboratory has been providing a service to re-refiners by evaluating in the laboratory their products, including feedstocks, base stocks (or base oils) and finished lubricating oils. This service has been available since 1967. In recent years it has been extended to oil reclaimers generally so that reclaimed industrial, and hydraulic oils have been examined as well (Refs. 1 and 2).

Regrettably, because of other commitments and absence of some equipment the evaluations have not been as thorough as desirable. For example, except for standard single cylinder engine tests on a number of re-refined engine oils in the late sixties, rig and field tests on finished products have not been done. Nevertheless, it is believed, that the tests that have been carried out have been valuable. Individual reports showing the test results were sent to respective re-refiners whose oils were tested.

Principally, re-refined oils obtained by the conventional acid/clay process (Appendix A) and samples sent by the re-refiner to the Fuels and Lubricants Laboratory were tested. However, a number of experimental base stock oils were examined from Canadian experimenters employing a variety of processes including vacuum distillation, hydro-treatment, solvents and chemicals.

Because the main thrust has been the evaluation of base stocks, leading to an accumulation of a good deal of data, this report gives only evaluations of base stocks from the acid/clay process. Samples were received from six Canadian re-refiners and one Indian re-refiner. Two of the Canadian re-refiners supplied a number of samples over a period of time (one over a period of 7 months, the other over a period of 10 years) enabling a measure of base stock consistency to be made. In addition one supplied a custom re-refined railroad base oil, identified as RR-F. It is also believed that the base stock supplied by re-refiner "B" was not obtained entirely from used engine oil drainings. Re-refiner "D" supplied sample FLO 74404, a partially re-refined oil.

To provide some reference data, five refined base stocks were also evaluated comprising three normally used for making engine oils, a white oil and a light high quality base oil used for making MIL-H-5606 hydraulic fluid. These data are also shown.

Testing was chosen to detect additives and contaminants commonly found in used oils, and to assess general quality. More specifically, testing, principally by Standard ASTM methods, included a check for (a) liquid contaminants, such as, glycol from antifreeze, and diluents from gasoline and diesel fuel; (b) solid contaminants, such as, lead decomposition products, soots, and road dusts; (c) oil decomposition products, such as acids and resins; (d) additives; (e) wear metals; (f) re-refinery process materials; and (g) response to anti-oxidants. In addition, many general physical and chemical tests, and hydrocarbon type characterizations were performed. Additional information on the significance of ASTM tests is given in ASTM STP7C (Ref. 3).

Results of testing, comparisons and comments are presented herein in tabulated form and in text. A full set of data is not shown in some instances because only small quantities of samples were received.

2.0 CANADIAN OIL RE-REFINERS

Samples of re-refined oil from the acid/clay process were received from six Canadian re-refiners. The following re-refiners submitted samples as indicated:

Forsythe Lubrication Associates, Breslau, Ontario
(now Breslube Enterprises)

Hub Oil Company Limited, Calgary, Alberta

Magnum Oil Company, Saskatoon, Saskatchewan

Schofield and de Vries Limited, Breslau, Ontario
(became Forsythe, now Breslube, see above)

Turbo Refineries, Edmonton, Alberta

Wills Oil Company, Vancouver, B.C.

The Canadian re-refiners are listed in alphabetical order which bears no relationship to the coding.

3.0 OBSERVATIONS, COMPARISONS AND COMMENTS

To simplify comparisons, comments do not apply to the RR re-refined oil, white oil and MIL-H-5606 base oil data unless specifically mentioned.

3.1 Contaminants

3.1.1 Liquid Contaminants

3.1.1.1 Glycol and Undissolved Water

No glycol was detected in eleven of twelve samples of re-refined oil tested. A quantity of 60 ppm was detected in one sample (Table 4). No undissolved water was detected in any of the samples.

3.1.1.2 Fuel Diluent

The test method used gives results showing trace amounts of fuel diluent in a number of the re-refined oil samples (Table 4), and also traces in some of the refined oils where no diluent is present. Accordingly, diluents are judged to be absent or substantially absent from all oils tested. This is further confirmed by the high flash points of all oils (Table 3). Notwithstanding, re-refined oils as shown by their distillation characteristics do have a more volatile front end than corresponding virgin oils (Tables 8, 15). This front end volatility of the re-refined oils is outside the normal boiling range of gasolines, however.

3.1.2 Solid Contaminants

Assuming that the preferred low total solids range is 0.5 to 3 mg/100 mL by ASTM D2276 based on the virgin oils, the best that the re-refined oils can do is to reach the upper limit of this range, although two oils out of twelve tested showed low values of 1.7 and 2.3 mg/100 mL (Tables 2, 3). Most re-refined oils showed total solids values of 3 to 5 mg/100 mL and two were as high as 12 mg/100 mL. The RR oil had a value of 47 mg/100 mL (Table 3). (A sample from a new batch currently under test showed a value of 6.4 mg/100 mL.)

3.1.3 WEAR AND ADDITIVE METALS

The wear and additive metals analyses along with the ash content tests (D482 or D874) are an important measure of the degree of re-refining. Among the data clearly are examples of good re-refining, indeed consistently good re-refining, by a Canadian re-refiner whose four samples since 1974 show 0.000% wt ash, comparable to virgin oils, and of fair re-refining where the ash is about 0.01% wt (Table 4). A realistic limit on ash content by ASTM D482, assuming reproducibility limitations in the

low ranges, could be 0.002 or 0.003% wt. Since ash and sulphated ash results were substantially alike, there is no real evidence that any substantial quantity of volatile metals escape during the ash test (Table 4). Nevertheless the limits could be based on the longer sulphated residue test, ASTM D874, if this was felt to be necessary.

3.1.4 Additives

Numerous standard tests can be used to detect the presence of additives although if the additives are present in only trace or practically nil amounts most tests are generally inadequate, unless efforts are made to test additives extracted from the oil, also a difficult operation. Such efforts were not made. Acid and base numbers, saponification numbers, IR analysis, elemental analysis, quantity of polar compounds and even the emulsion test will give a general measure of the presence of additives often even in trace amounts. A comparison of re-refined and virgin base oil chemical data of this type (Tables 4, 12) suggest that all the re-refined oils have a trace or a bare trace of additive or other polar compound, such as an oil or additive decomposition product. The presence of this trace material may or may not enhance the base stock. Conceivably in re-refiner B's oil it may have enhanced the anti-oxidant response while it degraded the demulsibility characteristics.

3.2 Distillation Range

Generally, the distillation range of re-refined oils is about 550 to 1050°F. This is broader than that noted for the virgin oils tested which is about 600 to 900°F. Both a gas chromatograph (gc) simulated distillation and a vacuum distillation test (Tables 8, 15) were performed in many instances. It is believed that the latter is more accurate when the end point exceeds about 1000°F. The former may show erroneously lower end point readings. Probably the gc distillation should be ended at 1000°F and per cent recovery reported at 1000°F.

3.3 General Physical Properties

Since the virgin oils are slightly lighter and less viscous than the re-refined oils a comparison of some of the properties related to oil viscosity and density is avoided. Some, however, are compared. Summarizing then, re-refined oil colour is darker, Ramsbottom carbon residue is higher, demulsibility is poorer, and odour is sharper than that of virgin oils. Copper strip corrosion is comparable. Foaming properties are mixed: some re-refined oils are inferior to virgin oils while one is far superior. Canadian re-refined base oil viscosities meet SAE 20 viscosity requirements, while the Indian oils meet SAE 30, and the Canadian RR oil meets SAE 40.

3.4 General Chemical Properties

Total acid numbers, total base numbers, total sulphur and total nitrogen contents, and saponification numbers of Canadian re-refined oils are low (Table 4). Although on the average not as low as those observed for virgin oils (Table 12) except for total base numbers which are more or less comparable. The Indian oils show very low total acid numbers and saponification numbers, comparable to virgin oil values. However, the total sulphur and one total nitrogen value are markedly higher (Tables 4, 12).

Only the RR oil shows a trace of strong acids possibly from the re-refining process (Table 4). (A current sample from another batch received recently for tests showed no strong acid number.) None of the other re-refined oils tested show a strong acid number.

3.5 Hydrocarbon Characterizations

Based on hydrocarbon types analysis by ASTM D2007, the aromatics for Canadian re-refined oils range from 18 to 22% weight (Table 4). This is slightly higher than for virgin oils tested which show an aromatics range of 16 to 18% weight (Table 12). Both the Indian and RR oils show a markedly higher aromatics content while the white oil and MIL-H-5606 base stock show a markedly lower aromatic content (Table 4). The structural group analysis data by carbon distribution (ASTM D3238)

tends to support the aromatics characterization directionally, except for the white oil data. ASTM D3238 analysis further indicates a lower naphthenic carbon content in the re-refined oils, while ASTM D2007 analysis indicates a higher polar content in these oils.

3.5.1 Infrared Analysis

Hydrocarbon type analysis of lube oil base stocks can be determined with infrared spectroscopy by examination of several different absorption bands which are characteristic of aromatic or paraffinic type materials. Carbonyl absorption bands can be indicative of polar type compounds present in the base stocks (Refer to Figure 1).

Alkyl absorptions at 2900, 1460, 1380, 970 and 720 cm^{-1} are characteristic of mineral oils, with the absorption band at 720 cm^{-1} being attributed to long chain paraffins, four or more methylene groups in a row. The spectrum of white oil, No. 1, is very characteristic of a paraffinic mineral oil.

The hydraulic base oil spectrum, No. 2, suggests it to be more naphthenic in character (ie: containing more cycloalkanes). This is evidenced by the typical paraffinic spectra with changes only in the 700-800 cm^{-1} wavenumber region. Part of the long chain methylene vibration at 720 cm^{-1} has been somewhat shifted to a higher frequency at 740 and 770 cm^{-1}. These shifts suggest either three or two methylene groups in a unit rather than four or more associated with the 720 cm^{-1} absorption. These absorptions along with the absence of any real aromatic absorptions at 1600 and 813 cm^{-1} suggest that the hydraulic base oil, although paraffinic does contain more cycloalkanes (naphthenics) than the white mineral oil.

Looking at the refined oil base stocks, absorptions at 1600 cm^{-1} in the No. 3 oil, and 1600, 813 cm^{-1} in the No. 4 oil are typical of C = C groups, suggesting aromatic compounds are present in these refined base stock oils and also suggesting that slightly more aromatic compounds are present in No. 4 oil than No. 3 oil.

The acid/clay re-refined oil spectrum, No. 5, is very similar to the spectrum of No. 4 virgin oil. The Indian acid/clay re-refined oil spectrum, No. 6, again is very similar to the spectra of the typical acid/clay oil and virgin oil. However the absorptions at 1600 and 813 cm^{-1} are stronger suggesting an higher aromatic content. These stronger aromatic absorptions are again seen in the spectrum of a railroad acid/clay re-refined oil, No. 7. A very weak absorption band at 1700 cm^{-1} seen in the railroad acid/clay oil is indicative of a carbonyl absorption C = 0; it often correlates with a total acid number determination.

3.6 Storage Characteristics

Storage characteristics were evaluated quite simply under three basic sets of conditions over a period of one year: (a) in a clear glass bottle in the sunlight (Table 9), (b) in a dark bottle and a metal can in a cupboard at room temperature, and (c) in the same containers but in a cold room at 40°F (Table 10).

Only the tests at room temperature in a cupboard had no effect on any of the oils, re-refined and virgin alike. All the oils were clear and free from sediment after one year of storage except for the RR oil which was dark before storage and difficult to see through at anytime.

Testing in daylight had a marked effect on the re-refined oils only; a white flocculent precipitate tended to appear after about 3 to 6 months storage. The two Indian oils showed the least amount of precipitate. Virgin oils tested showed no precipitate. (Further recent testing of other virgin base stocks shows that some do indeed develop precipitation, but unlike the flocculent precipitate of the re-refined oils.)

Testing at 40°F tended to generate a whitish precipitate in re-refined oils; nothing was generated in virgin oils. Because of the high cloud point of the Indian oils (+45 and +50°F), the oils clouded immediately at 40°F making it impossible to assess their storage stability at 40°F.

No attempts were made to identify the flocculent suspensions generated in the daylight and at low temperature.

3.7 Oxidation Stability and Response to Antioxidants

3.7.1 Oxidation Stability

A number of oxidation stability tests were performed on the base stocks, principally as an academic exercise because base stocks are now seldom used alone, and because their response to antioxidants is often independent of basestock oxidation stability.

The following tests were performed on a number of oils:

- British Air Ministry Oxidation Test (IP48/67)
- ERDA Oxidation Test (Combination D943/D2274)
- CIGRE Oxidation Test (IP306)
- L-38 Bearing Corrosion and Oxidation Stability
- Oxidation Stability (ASTM D943).

These results were obtained on both re-refined and virgin oils:

3.7.1.1 British Air Ministry Oxidation Test (IP48/67)

Oil	Refiner/Re-refiner	Test	Viscosity Ratio at 100°F, cSt/cSt, Oxid./New	Rams. Carb. Res. Incr. % wt.
FLO75006	Re-refiner "A"	(1)	1.44	0.93
		(2)	1.36	0.81
FLO75007	Re-refiner "A"		1.40	0.34
FLO74402	Re-refiner "D"	(1)	1.40	0.49
		(1)	1.43	0.50
FLO74404	Re-refiner "D"	(1)	2.21	1.97
		(2)	1.81	1.48
FLO74335	Re-refiner "F"	(1)	1.42	0.50
		(2)	1.52	0.54
FLO74396	Re-refiner "C"	(1)	1.27	0.72
		(2)	1.64	1.30
		(3)	1.66	1.35
FLO74336	Refiner "I"	(1)	1.31	0.35
		(2)	1.39	0.48
FLO74337	Refiner "II"	(1)	1.96	0.99
		(2)	1.93	0.97

Old CGSB Specification Limits

3-GP-51 (Engine Oil)	2.00 max	1.0 max
3-GP-100B (Aviation Engine Oil)	1.75 max	1.0 max

3.7.1.2 Summary (B.A.M., IP48)

Excluding the partially re-refined oil, FLO74404, which shows the poorest oxidation stability in this high temperature test, the re-refined oils are substantially alike meeting all old CGSB engine oil requirements, except for one of the oils which fails to meet the carbon residue increase requirement of 1.0 max.

One of the virgin oils appears slightly superior to the re-refined oils; the other is inferior.

3.7.1.3 ERDA Oxidation Test, (Mod. D943/D2274)*

	Re-refined				Virgin	
Property (Changes)*	FLO 74335	FLO 74396	FLO 74402	FLO*** 74404	FLO 74336	FLO 74337
Filt. Res., mg/200mL	5.9	2.5	7.6	53.4	81.2	1.5
Adh. Res., mg/200mL	6.6	3.6	8.5	5.3	7.2	2.3
Colour	>5	>5	>4	(D8.0)**	2	3
Viscosity @100°F, cSt	2.5	2.4	1.4	7.8	2.5	2.1
Viscosity @210°F, cSt	0.2	0.2	0.1	0.5	0.2	0.2
IpH Reading	-3.8	-2.9	—	—	-2.5	-0.3
Total Acid No.	0.7	0.6	0.4	0.4	2.4	0.3
Total Base No.	-0.1	0.0	0.0	0.0	-0.4	0.0
Saponification No.	0.6	1.6	-0.1	1.4	5.3	-0.2
Pentane Insol. % wt.	0.04	-0.02	0.01	0.19	0.04	0.07

* The values shown are changes, ie., oxidized value minus new oil value.
** Both the new oil and oxidized oil colour D8.0.
*** Partially re-refined.

3.7.1.4 Summary (ERDA)

The fully re-refined oils tend to have substantially similar ERDA oxidation characteristics. Considering that an antioxidant is absent the results seem low. Only the partially re-refined oil shows elevated values, ie., filterable residue, viscosity and pentane insolubles.

Again the virgin oils split in performance as they did for the BAM Test except that the relative performances of the two are reversed. Whereas FLO74337 oil performed well and FLO74336 oil performed poorly in the 200°F ERDA test the reverse is true in the high temperature 400°F BAM Test.

3.7.1.5 CIGRE Oxidation Test IP306

	Re-refined				Virgin	
	FLO 74335	FLO 74396	FLO 74402	FLO* 74404	FLO 74336	FLO 74337
New Oil						
Total Acid No.	0.1	0.1	0.1	0.1	0.0	0.1
T.O.P.	0.1	0.0	0.0	0.0	0.0	0.0
Oxidized Oil — No Catalyst						
Total Acidity	0.2	0.3	0.1	0.8	17.8	0.0
Total Sludge, % wt.	0.0	0.0	0.0	0.0	0.9	0.0
T.O.P.	0.1	0.1	0.1	0.3	6.6	0.0
Oxidized Oil — Solid Catalyst						
Total Acidity	4.3	3.1	2.2	5.6	29.9	1.8
Total Sludge, % wt.	0.7	0.4	0.3	1.5	8.0	0.2
T.O.P.	2.4	1.4	1.0	3.3	17.6	0.7
Oxidized Oil — Soluble Catalyst						
Total Acidity	4.0	—	—	—	28.8	5.4
Total Sludge, % wt.	0.6	—	—	—	6.5	0.4
T.O.P.	1.9	—	—	—	15.7	2.2

* Partially re-refined.

3.7.1.6 Summary (CIGRE)

This test appears to convey about the same information, irrespective of catalyst condition, as the ERDA Test, ie., all re-refined oils are substantially alike and good, except for the partially re-refined oil, which has undergone slightly more degradation. The two virgin oils split in performance and behaved just as they did in the ERDA Test; FLO74337 oil showed superior performance to the re-refined oils while FLO74336 oil showed vastly inferior performance.

3.7.1.7 L-38 Bearing Corrosion and Oxidation Stability Test, FLO74485, (Re-Ref. "D") (After 10 Hours in Test)

Viscosity Inc. at 210°F, %	61.7
Total Acid No. Inc.	13.0
Bearing Wt. Loss, mg (Total)	429.5
Hydrocarbon Types (Oxid. Oil) (D2007, % wt.)	
Saturates	52.4
Aromatics	19.4
Polar Compounds	28.2
Infrared Analysis	Heavy Peaks at 1710 cm^{-1}

3.7.1.8 ASTM D943 Oxidation Test

Re-refined and virgin base stocks alike broke down within 168 hours.

3.7.2 Response to Antioxidants (Tables 6, 7, 13, 14)

Of much more significance than oxidation stability testing of uninhibited base stock is the testing of inhibited base stock to assess its response to antioxidant. ASTM D943 was the test selected more or less arbitrarily because even though long in test time it does not require a disproportionate amount of operator time. Furthermore it is a well-recognized test yielding response of base stocks destined for formulations for hydraulic fluid and steam turbine oil use. The two additives used were zinc dialkyldithiophosphate and 2,6-ditertiary-butyl para-cresol. It was hoped that at least some comparative data would be generated.

The D943 oxidation lifetime data of Canadian oils (with additive) tend to suggest some correlations to hydrocarbon types, viscosity, sulphur, total acid number and carbon residue, although it is not known precisely which ones are operative in each instance. Unfortunately, the Indian oil data contradicts some of these apparent correlations, e.g., high aromatics and high sulphur contents have not hurt Indian oil oxidation lifetimes. Obviously, any real correlations would have to be based on more detailed analyses of hydrocarbon types by a method other than ASTM D2007. For example, polar and/or sulphur compounds in the base stock may enhance or inhibit the additive role. Just knowing the total amounts of one or the other is not enough; the actual active compounds may have to be identified.

4.0 SUGGESTED LIMITS FOR WELL RE-REFINED CANADIAN BASE OILS

Some suggested limits for various properties were indicated from this study and are given in Table 1. Data taken from the U.S. Army/Environmental Protection Agency study in the U.S. (Ref. 4) were also considered in arriving at some of the suggested limits. While some rigidity is needed to control the levels of liquid and solid contaminants and hence to retain limits governing them, more flexibility must be considered in respect to other properties due to the changing crude oil picture and changing machine and engine technology leading to oil products of differing compositions. Some of the testing could be eliminated to reduce the burden on the re-refiner. For example, a control on the ash content might suffice to control the levels of various metallic elements for which tests may not need to be done. Depending on the use to which the base stock is put it may even be desirable to have a number of grades of base stock.

5.0 CONSISTENCY OF RE-REFINED BASE STOCK

A measure of batch-to-batch consistency was obtained when three re-refiners submitted samples on different occasions. One supplied seven samples over a span of 10 years and another supplied six samples over a span of six months. Very good consistency coupled with very good quality were obtained particularly by re-refiner "F". Ranges are given in Table 2 while individual data are given in Appendices C and D.

6.0 CONCLUSIONS

Canadian re-refined base stocks when well-re-refined by the acid/clay process have excellent properties including a good response to anti-oxidants and a high degree of cleanliness. This is shown from a study of base stocks (19 samples) received from six Canadian re-refiners over a span of 10 years.

Since traces of additives and/or polar compounds do remain, the quality of the base stocks is judged to be very slightly inferior to that of comparable virgin refined base stocks. This statement is not intended to imply that formulations containing re-refined base stocks are inferior; they may in fact be superior. Only testing in the laboratory and field of fully formulated oils containing re-refined base stock would truly indicate quality.

Some useful suggested specifications limits for various properties were obtained as well as a good indication of batch to batch consistency.

7.0 RECOMMENDATION

ASTM standards are absent in some areas and some ASTM standards are inadequate. For example, good ASTM standards are needed for detecting metals and non-metals in low ranges. Ash (D482) and sulphated ash (D874) methods could be extended to lower levels of detectability. Qualitative tests for appearance and odour would be useful. An ASTM or CGSB standard specification could even be developed for re-refined base stock quality, if only to limit the levels of used oil contaminants, additives and process materials. There could even be a number of grades.

8.0 ACKNOWLEDGEMENTS

The authors gratefully acknowledge the co-operation provided by the re-refiners particularly in supplying samples. They also appreciate with much gratitude the analytical data provided by Dr. E. Furimsky of the Department of Energy, Mines and Resources, and by a number of commercial laboratories.

9.0 REFERENCES

1. Strigner, P.L. — *Used Oil Re-refining.* NRC, DME Newsletter on Manufacturing Developments, Vol. 7, No. 1, September 1975.

2. *Waste Oil — The Second Time Around.* Science Dimension, NRC, Vol. 7, No. 5, 1975.

3. Boldt, K.; Hall, B.R. — *Significance of Tests for Petroleum Products.* ASTM STP 7C.

4. Frame, E.A.; Bowen, T.C., Jr. — *U.S. Army/Environmental Protection Agency Re-refined Engine Oil Program.* AFLRL Report No. 98.

TABLE 1

SUGGESTED LIMITS FOR WELL RE-REFINED CANADIAN BASE OILS
(Acid/Clay Processed from Service Station Drainings)

TEST	METHOD	SUGGESTED LIMITS	
	PHYSICAL TESTS		
Gravity and Viscosity			
API Gravity at 60°F	D287	28.5-30.5	
Viscosity Index	D2270	95-115	
Viscosity at 100°C, cSt	D445	7.0-9.4	
Solid Contaminants and Ash			
Trace Sediment, % V	D2273	0.001	max.
Solids, mg/100 mL	D2276	5	max.
Precipitation No.	D91	0.001	max.
n-Pentane Insol., % W	D893/B	0.02	max.
Ramsbottom Carbon Residue, % W	D524	0.20	max.
Ash or Sulfated Ash, % W	D482 or D874	0.005	max.
Liquid Contaminants			
Flash Point, °F	D92	400	min.
Dilution, % V	D3525	0.05	max.
Dilution, % V	D322	Nil	
Glycol	D2982	Nil	
Water, % V	D95	Nil	
Workmanship			
Appearance	Visual	Clean, clear	
Odour	Smell	Characteristic	
Colour	D1500	4.0	max.
Miscellaneous			
Copper Strip Corrosion, 3 hrs at 100°C	D130	No. 1	
Aniline Point, °F	D611	220-226	
Cloud Point, °F	D2500	+24	max.
Pour Point, °F	D97	+15	max.
Distillation Range, °F	D2887		
I.B.P.		550	min.
F.B.P.		1100	max. (1)
	CHEMICAL TESTS		
Neutralization and Saponification Numbers			
Total Acid No.	D664	0.20	max.
Total Base No.	D664	0.10	max.
Strong Acid and Strong Base No.	D664	Nil	
Initial pH Reading	D664	6-8	
Saponification No.	D94	1.0	max.
Hydrocarbon Types	D2007		
Saturates, % W		75-80	
Aromatics, % W		20-22	
Polar Compounds, % W		2.5	max.
Metals and Silicon			
Calcium, Barium, ppm	AA	10	max. (2)
Zn, Pb, Mg, Cu, Cr, Fe, Na, ppm	AA	5	max. (2)
Silicon, ppm	AA	10	max.
Non-Metals			
Sulfur, % W	D129, D1552	0.20	max.
Nitrogen, ppm	?	100	max.
Chlorine, ppm (3)	D1317	50	max.
Phosphorus	D1091	15	max.
Oxygen	?	0.60	max.
Response to Anti-oxidant			
Oxidation with 0.3% W DBPC, hrs	D943	504	min.

Notes: (1) Or in lieu of 1100°F max. temperature use a limit on the residue at 600°C of 5% W. max.
(2) Each element.
(3) Includes bromine; ASTM D1317 measures halogens. If a new standard test detecting lower levels becomes available, the chlorine limit could be lowered to say 20 ppm or lower.

TABLE 2

CONSISTENCY OF RE-REFINED BASE STOCK

	RE-REFINER "E"	RE-REFINER "F"
No. of Samples	6	6
Period, Years (Years samples received)	1/2(1967)	10(1968-78)
PHYSICAL PROPERTIES		
Gravity and Viscosity		
API Gravity (D287)	28.8-29.5	29.1-29.9
Viscosity at 210°F (D445), cSt.	8.6-9.4	7.3-8.2
Viscosity Index (D2270)	101-104	97-102
Solid Contaminants and Ash		
Trace Sediment (D2273), % V	0.000-0.004	0.000-0.001
Solids (D2276), mg/100 mL	–	1.7-5.3
Ash (D482), % W	0.005-0.03	0.000
Rams. Carb. Res. (D524), % W	0.12-0.24	0.11-0.17
Liquid Contaminants		
Flash Point (D92), °F	440-460	390-440
Dilution (D322), % V	Nil	Nil
Glycol (D2982)	Nil	Nil
Miscellaneous		
Colour (D1500)	L3.5-L6.5	L2.0-3.5
Copper Strip Corrosion, 3 hrs at 100°C	No. 1	No. 1
Aniline Point (D611), °F	225-227.5	222-226
CHEMICAL PROPERTIES		
Neutralization and Saponification Numbers		
Total Acid No. (D664)	0.12-0.37	0.04-0.20
Total Base No. (D664)	0.00-0.01	0.00-0.09
Strong Acid No. (D664)	Nil	Nil
Saponification No. (D94)	–	0.12-1.0
Initial pH Reading (D664)	–	6.0-8.3
Hydrocarbon Types (D2007)		
Saturates, % W	–	75.9-78.0
Aromatics, % W	–	20.3-22.2
Polar Compounds, % W	–	1.6-2.2
Miscellaneous		
Sulfur (D129), % W	0.09-0.22	0.08-0.21
Nitrogen, ppm (2)	–	5-33
Molecular Weight (D2502)	479-496	448-475
Metallic Elements (AA)	–	<5 (1)

Notes: (1) Each Element. Elements tested were: A1, Ba, Ca, Cr, Cu, Fe, Mg, Pb, Si and Zn.
(2) Determined by Canadian Department of Energy, Mines and Resources by microcoulometry.

TABLE 3

PHYSICAL TEST DATA

OF

ACID/CLAY PROCESS — RE-REFINED BASE STOCK OILS

RE-REFINER	A	INDIAN(1)	B	C	D(2)	E(2)	F(2)	RR-F
ASTM Colour (D1500)	5.5	3.5	2.0	3.0	4.5	4.5	3.0	D8.0
API Gravity at 60°F (D287)	—	27.5	30.2	30.2	29.7	29.1	29.6	26.1
Specific Gravity at 60/60°F (D287)	—	0.8899	0.8751	0.8751	0.8781	0.8813	0.8785	0.8978
Flash Point, °F (D92)	440	425	420	—	395	454	418	480
Copper Strip Corrosion (3 hrs @ 212°F) (D130)	—	No. 1a	No. 1b	No. 1a	—	No. 1	No. 1a(3)	No. 1a
Cloud Point, °F (D2500)	—	+45	+18	+12	—	+16	+19	+20(8)
Pour Point, °F (D97)	+5	+45	+10	+ 5	—	-24	- 2.5	0
Viscosity at 100°F, cSt (D445)	72.25	103	49.93	57.18	59.48	74.48	60.03	187.5
Viscosity at 210°F, cSt (D445)	8.88	11.20	7.18	7.66	7.80	8.98	7.64	14.48
Viscosity Index (D2270)	105	103	113	107	105	103	100	79
Precipitation No. (D91)	—	0.001	0.000	< 0.001	0.002	—	0.000	< 0.001
n-Pentane Insol., %/wt (D893/A)	—	0.02	0.00	0.07	0.07	—	0.02	0.01
Total Solids, mg/100 ml (D2276)	11.8&2.3	4.7&3.1	4.9	3.3	12.8	—	3.30	46.6
Trace Sediment, % Vol (D2273)	0.005	0.004	0.001	0.000	0.05	< 0.001	0.001	0.001
An-Oil-Izer	—	+ 1.5	- 5.5	- 4.8	- 3.3	—	- 3.0	+ 2.0
Appearance (Visual)	Note(4)	Note(4)	Note(4)	Note(4)	Note(5)	Note(4)	Note(4)	Note(9)
Odour	—	Note(6)	Note(7)	Note(7)	Note(7)	Note(7)	Note(7)	Note(7)
Ramsbottom Carbon Residue, % wt (D524)	0.17	0.26	0.14	0.14	0.19	0.18	0.14	0.27
Viscosity-Gravity Constant (D2501)	—	0.84	0.82	0.81	0.81	0.84	0.83	0.82
Modecular Weight (Est) (D2502)	490	515	468	462	470	485	460	520
Foaming (D892)								
Tendency at 75°F, ml	—	515	30	—	—	544	428	0
Tendency at 200°F, ml	—	55	20	—	—	69	38	50
Tendency at 75°F, after 200°F, ml	—	455	30	—	—	508	435	0
Stability at all conditions, ml (After 10 min)	—	80-0-80	0	—	—	166-1-87	7-0-9	0
Emulsion Characteristics (D1401)	—	40-39-1(20)	22-0-58(60)	—	—	—	33-37-3(35)	0-0-80(60)
Refractive Index (20°C)	1.4831	1.4920	1.4829	—	1.4830	—	1.4837	1.4942

Notes: (1) Average of 2 samples.
(2) Average of 4 to 7 samples. See respective tables in the Appendix.
(3) One sample was No. 2b.
(4) Clean, clear and free from visible contaminants.
(5) One sample was hazy with some settled black sediment, another showed a trace of black sediment on standing.
(6) Fairly sweet odour — not characteristic of re-refined base stocks produced by this method.
(7) Odour characteristic of re-refined base stocks produced by this method — slightly unpleasant — but not like used oil.
(8) Approximate Cloud Point; entire sample tended to get cloudy at one time, possibly due to the presence of trace additive.
(9) Sample too dark to make any visual appearance test.

TABLE 4

CHEMICAL TEST DATA

OF

ACID/CLAY PROCESS — RE-REFINED BASE STOCK OILS

RE-REFINER	A	INDIAN(1)(8)	B	C	D(2)	E(2)	F(2)(8)	RR-F
Sulphated Residue, % wt (D874)	—	0.003	0.002	0.008	0.001(6)	—	—	0.004
Ash, % wt (D482)	0.005	0.002	—	0.014	0.002(6)	0.02	0.000	0.002
Total Sulphur, % wt (D129)	—	0.91	0.11	0.10	—	0.15	0.13	0.66
Nitrogen (ng/μL) (*Microcoulometer-Dohrmann*)(3)	—	131(4)	10	—	—	—	20	46
Aniline Point, °F (D611)	—	228.3	224.1	222.4	223.1	225.8	223.1	226.8
Hydrocarbon Types (D2007)								
Saturates, % wt	75.0	65.5	79.5(7)	77.2	75.5	—	77.1	64.5
Aromatics, % wt	21.1	32.4	17.8(7)	19.6	21.8	—	21.0	33.0
Polar Compounds, % wt	3.9	2.1	2.7(7)	3.2	2.6	—	1.9	2.5
Strong Acid No. (D664)	Nil	Nil	Nil	Nil	—	Nil	Nil	0.07
Total Acid No. (D664)	0.21	0.02	0.06	0.24	0.24	0.23	0.11	0.27
Total Base No. (D664)	—	0.00	0.02	Nil	0.00	0.00	0.01	0.00
IpH Reading (D664)	4.5	6.6	5.5	—	—	—	7.1	2.5
Crankcase Dilution, % Vol (D322)	—	0.01	0.4	—	—	—	Nil	Nil
Dilution by GC, % wt (D3525)	—	0.03	0.2	0.03	—	—	Nil	Nil
Glycol Content (D2982)	—	Nil	Nil	TRACE(5)	—	Nil	Nil	Nil
Saponification No. (D94)	—	0.25	0.96	1.12	1.21	—	0.66	0.48
Carbon Distribution & Structural Group Analysis, n-d-m Method (D3238)								
% C_A	—	8.7	6.8	—	—	—	5.9	8.8
% C_N	—	24.4	24.6	—	—	—	27.8	27.1
% C_P	—	66.9	68.6	—	—	—	66.3	64.1
R_A	—	0.55	0.38	—	—	—	0.33	0.55
R_N	—	1.96	1.77	—	—	—	1.97	2.25
R_P	—	2.50	2.15	—	—	—	2.30	2.80
Carbon, % wt (240 Analyzer)	—	—	—	—	—	—	86.6(3)	—
Hydrogen, % wt (240 Analyzer)	—	—	—	—	—	—	13.5(3)	—

Notes:
(1) Average of two samples.
(2) Average of four to seven samples. See respective tables in the Appendix.
(3) Performed by another laboratory; one sample only tested for carbon and hydrogen.
(4) The two oils had markedly different values of 13 and 348 ng/μL.
(5) Estimated at 60 ppm Glycol.
(6) Average of three samples; the fourth sample had a value of 0.176% wt sulphated residue (partially re-refined sample).
(7) Identical results from a duplicate determination.
(8) Total chlorine determinations on a few samples by ASTM D1317 showed <10 ppm for the Indian oils and 25 ppm for "F". Tests by a commercial laboratory using neutron activation showed 1.2 ppm for the Indian oils and 19 ppm for "F". Because D1317 includes all halogens the figure of 25 ppm may include bromine, so that, in fact, the agreement between the two techniques is very close.

TABLE 5

METALLIC ELEMENTS (PPM)

IN

ACID/CLAY PROCESS — RE-REFINED BASE STOCK OILS

RE-REFINER	A(1)	INDIAN(2)	B(3)	C(4)	D(5)	F(6)	RR-F
Zinc	2	<1	8	3	< 1(7)	<1	<1
Sodium	2	—	—	1	—	—	—
Silver	<0.1	—	—	<0.1	< 1	<1	<1
Copper	<1	<1	<1	0.6	< 1	<1	<1
Calcium	5	<1	12	18	—	<1	<1
Aluminum	2	<1	<1	9	4	<1	<1
Nickel	<1	—	—	<0.1	< 1	<1	<1
Iron	2	<1	<1	18	<10(8)	<1	<1
Silicon	8	<5	<5	5	9	1	<1
Tin	0.5	—	—	0.2	< 1	1	<1
Lead	12	<1	<1	5	<10(9)	<1	<1
Chromium	1	<1	—	0.2	< 1	<1	<1
Magnesium	5	<1	<1	2	< 5	<1	<1
Manganese	0.2	—	—	0.6	—	—	—
Phosphorus	2	—	—	3	—	—	—
Boron	<0.1	—	—	<0.1	< 1	<1	<1
Barium	10	<1	<1	6	—	<1	<1
Titanium	0.05	—	—	<0.1	—	—	—
Cadmium	<0.1	—	—	—	—	—	—
Molybdenum	0.05	—	—	—	—	—	—

Notes: (1) Determined by Emission Spectroscopy in another laboratory.
(2) Determined by Atomic Absorption — Average of two samples.
(3) Determined by Atomic Absorption.
(4) Determined by Emission Spectroscopy in another laboratory — Average of three samples.
(5) Determined by Emission Spectroscopy in another laboratory — Average of four samples (See Note 6, Table 4) (See Table B2 in Appendix B).
(6) Determined by at least one of three methods: (a) Emission Spectroscopy, (b) Atomic Absorption or (c) by ASTM complexometric method Page 1016, Part A, 1965 (Calcium, Barium and Zinc only) Average of six samples.
(7) One sample >1000 ppm (See Note 6, Table 4). Result excluded.
(8) One sample 23 ppm (See Note 6, Table 4). Result excluded.
(9) One sample 350 ppm (See Note 6, Table 4). Result excluded.

TABLE 6

RESPONSE TO ANTIOXIDANTS (ASTM D943-54)(1)

ACID/CLAY PROCESS — RE-REFINED BASE STOCK OILS

TREATED WITH 0.3% 2,6-DITERTIARY-BUTYL PARA-CRESOL

RE-REFINER	A	INDIAN(2)	B	C	F(3)	RR-F
Test Duration	Total Acid Number D974, mg KOH/gm					
New Oil + Additive	0.16	0.02	0.01	0.17	0.09	0.28
168 hrs	0.37	0.07	0.00	0.20	0.47	1.40
336	3.07	0.06	0.06	0.18	0.42	14.02
504	—	0.22	0.23	0.19	16.51	—
672	—	0.44	0.40	0.33	—	—
840	—	0.79	0.69	10.36	—	—
1008	—	1.22	1.10	—	—	—
1176	—	4.64	1.64	—	—	—
1344	—	—	1.91	—	—	—
1512	—	—	2.20	—	—	—
1680	—	—	—	—	—	—

Notes: (1) 3 ml of sample drawn weekly.
(2) Average of two samples: 1176 and 1344 hours.
(3) Average of four samples.

TABLE 7

RESPONSE TO ANTIOXIDANTS (ASTM D943-54)(1)

OF

ACID/CLAY PROCESS — RE-REFINED BASE STOCK OILS

TREATED WITH 0.5% ZINC DIALKYLDITHIOPHOSPHATE

RE-REFINER	INDIAN(2)	B	F(3)	RR-F
Test Duration	Total Acid Number D974, mg KOH/gm			
New Oil & Additive	0.69	0.42	0.63	0.89
168 hrs	0.12	0.24	0.29	0.76
336	0.10	0.16	0.21	1.22
504	0.33	0.08	0.28	12.17
672	0.61	0.02	0.35	—
840	0.68	0.08	0.46	—
1008	10.04	0.21	9.59	—
1176	—	0.43	—	—
1344	—	0.49	—	—
1512	—	1.30	—	—
1680	—	1.69	—	—
1848	—	2.31	—	—
2016	—	3.55	—	—

Notes: (1) 3 ml of sample drawn weekly.
(2) Average of two samples: 840 and 1344 hours.
(3) Average of four samples.

TABLE 8

SIMULATED DISTILLATION DATA (MODIFIED D-2887)[1]

OF

ACID/CLAY PROCESS — RE-REFINED BASE STOCK OILS

RE-REFINER	INDIAN[2]		B	F[3]	RR-F[4]
Initial Boiling Point, °F	570	554	542	583	669(706)
5% Distilled, °F	650	638	687	669	781(793)
10% Distilled, °F	695	680	711	700	813(821)
20% Distilled, °F	758	751	740	730	840(833)
30% Distilled, °F	820	786	763	754	865(858)
40% Distilled, °F	860	809	783	776	875(885)
50% Distilled, °F	885	830	794	791	891(902)
60% Distilled, °F	916	851	820	810	906(925)
70% Distilled, °F	942	871	845	827	916(948)
80% Distilled, °F	965	912	875	846	927(975)
90% Distilled, °F	990	939	914	880	939(1036)
95% Distilled, °F	1004	954	950	897	942
Final Boiling Point, °F[5]	1015	970	1018	924	962(1063)

Notes:
(1) Final column Temp. 350°C.
(2) Two samples.
(3) Average of four samples.
(4) Vacuum distillation data in parenthesis; recovery is 96%.
(5) Tailings at the end; the final boiling point should be higher; note vacuum distillation data for RR-F.

TABLE 9

EFFECT OF ONE YEAR'S STORAGE IN SUNLIGHT

STORAGE ↓ / RE-REFINER ➡	INDIAN(1)		B	C	F(2)		RR-F
	Re-refined Oils						
	ASTM Colour (D1500)						
Start	< 4.0	3.0	2.0	3.0	3.5	2.5	D8.0
One Year	4.5	5.5	3.5	< 3.5	4.0	D8.0	8.0
	Total Sediment (D2273), % Vol.						
Start	0.004	0.003	0.000(3)	< 0.001(3)	0.000	0.000(4)	0.001
One Year	0.002	0.002	0.000(3)	< 0.001(3)	< 0.001	1.20 (4)	< 0.001
	Appearance						
Start	Clear	Clear	Clear	—	Clear	Dark	Dark
One Year	Trace(5) Flock	Trace(5) Flock	Trace(6) Sediment	—	Flock(7)	Flock(8)	Sediment(9)

STORAGE ↓ / REFINER OR OIL ➡	X	Z	WHITE OIL	HYDRAULIC BASE OIL
	Refined Oils			
	ASTM Colour (D1500)			
Start	< 0.5	< 0.5	0.0	0.0
One Year	< 2.6	2.0	0.0	< 0.5
	Total Sediment (D2273) % Vol.			
Start	< 0.001	0.000	0.000	0.000
One Year	< 0.001	< 0.001	0.0015	0.003
	Appearance			
Start	Clear	Clear	Clear	Clear
One Year	Clear	Clear	Clear	Clear

Notes:
(1) Two samples received a year apart.
(2) Two samples received two years apart.
(3) Precipitation number by ASTM D91.
(4) Precipitation number at start was 0.000; after one year, 0.20.
(5) Trace of flocculent material appeared after 6 months of storage; it did not increase in amount after one year of storage although some of it settled. Generally the Indian oils had far less flocculent precipitation than most Canadian re-refined oils.
(6) No flocculation, only a trace of sediment.
(7) A thick flocculent suspension appeared after three months storage. After one year of storage there was a good deal of flocculent matter and sediment. Much of it was redissolved upon warming to room temperature.
(8) Oil darkened considerably and showed a trace of haze at the bottom after three months. Colour was 8.0 and precipitation No. was 0.04 after six months; due to the dark colour it was impossible to observe for precipitation although a trace of sediment was observed at the bottom. Much settled matter and matter clinging to the walls of the sample bottle were observed after one year storage.
(9) Due to dark colour too difficult to observe for flocculation. Some black sediment had settled out.

TABLE 10

EFFECT OF ONE YEARS' STORAGE IN COLD ROOM (40°F)

STORAGE ⬇	RE-REFINER ➡	INDIAN(1)	B	F(2)	RR-F
		Appearance at 40° F — Re-refined Oils			
Start		Clear	Clear	Clear	Dark
After One Year		Cloudy due to high cloud point (+45° F)	Cloudy but no precipitation	Flocculent suspension and sediment (first appeared after two months)	Difficult to observe precipitation due to dark colour; some black sediment noted though.

STORAGE ⬇	REFINER OR OIL ➡	X	Y	WHITE OIL	HYDRAULIC BASE OIL
		Appearance at 40° F — Refined Oils			
Start		Clear	Clear	Clear	Clear
One Year		Clear	Clear	Clear	Clear

Notes: (1) Two samples examined. Samples received a year apart.
(2) Several samples examined. Samples received over a span of several years.

TABLE 11

PHYSICAL TEST DATA

OF

REFINED BASE STOCKS

BASE STOCK	X(1)	Y	Z(2)	WHITE OIL	HYDRAULIC BASE
ASTM Colour (D1500)	L0.5	L0.5	L0.5	0.0	0.0
API Gravity at 60°F (D287)	32.2	31.9	29.9	29.7	35.0
Specific Gravity at 60/60°F (D287)	0.8647	0.8660	0.8765	0.8778	0.8499
Flash Point, °F, (D-92)	420	435	425	405	220
Copper Strip Corrosion (D130)	No. 1a	No. 1b	No. 1a	No. 1b(3)	No. 1a
Cloud Point, °F (D2500)	+4	0	+5	-14	< -70
Pour Point, °F (D97)	+3.5	-5	+9	-15	< -70
Viscosity at 100°F, cSt (D445)	32.57	38.57	34.75	13.86	3.53
Viscosity at 210°F, cSt (D445)	5.18	5.75	5.28	3.01	1.30
Viscosity Index (D2270)	96	97	91	70	—
Precipitation No. (D91)	0.001	0.000	0.000	0.000	0.000
n-Pentane Insol., % wt. (D893/A)	0.02	0.04	0.02	0.00	0.00
Total Solids mg/100 ml (D2276)	3.4	1.1	2.1	0.44	1.08
Trace Sediment, % Vol. (D2273)	0.001	0.000	0.000	0.000	0.000
AN-OIL-IZER	-7	-6.9	-5.7	-9.0	-12.0
Appearance (Visual)	Clear(4)	Clear(4)	Clear(4)	Clear(5)	Clear(5)
Odour	Note(6)	Note(6)	*Note*(6)	*Note*(6)	*Note*(6)
Ramsbottom Carbon Residue, % wt. (D524)	0.07	0.05	0.07	0.07	0.07
Viscosity-Gravity Constant (D2501)	0.81	0.81	0.82	0.84	Note(7)
Molecular Weight (Est) (D2502)	373	418	395	300	Note(7)
Foaming (D892)					
Tendency at 75°F, ml	320	—	365	460	30
Tendency at 200°F, ml	20	—	35	20	0
Tendency at 75°F after 200°F, ml	340	418	395	400	30
Stability at all conditions, ml (after 10 min.)	0	—	0	0	0
Emulsion Characteristics (D1401)	40-40-0(7.5)	—	40-40-0(5)	40-40-0(5)	40-40-0(5)
Refractive Index (20°C)	1.4761	1.4763	1.4812	1.4810	1.4638

Notes: (1) Average of two samples.
(2) Average of three samples.
(3) Trace Magenta (No. 2a), noted at end of strip. Several repeat tests performed *all showed the same phenomenon.*
(4) Clean, crystal clear and free from visible impurities.
(5) Clean, clear and water white in colour.
(6) Characteristic, pleasant oily refined oil odour.
(7) Unable to determine due to the very low viscosity and density of the oil.

TABLE 12

CHEMICAL TEST DATA OF REFINED BASE STOCK OILS[14]

BASE STOCK	X[1]	Y	Z[2]	WHITE OIL	HYDRAULIC BASE
Sulphated Residue, % wt. (D874)	0.000	0.000	0.000	0.000	0.000
Ash, % wt. (D482)	0.000	0.000	0.000	0.000	0.000
Total Sulphur, % wt. (D129)	0.08	0.000	0.05	0.00	0.01
Nitrogen (ng/μL) (Microcoulometer-Dohrmann)[3]	36[4]	—	18	15	1
Aniline Point, °F (D611)	220.3	225.0	210.9	232.7	167.7
Hydrocarbon Types (D2007)					
Saturates, % wt.	84.6	84.4	80.5	95.1	92.4[12]
Aromatics, % wt.	14.7	14.8	18.3	4.0	6.6[12]
Polar Compounds, % wt.	0.7	0.8	1.2	0.9	1.1[12]
Strong Acid No. (D664)	Nil	Nil	Nil	Nil	Nil
Total Acid No. (D664)	0.07	0.00	0.05	0.01	0.03
Total Base No. (D664)	0.02	0.00	0.00	0.00	0.00
IpH Reading (D664)	6.9	6.8	7.4	6.6	6.7
Crankcase Dilution, % Vol (D322)	Nil	—	Nil	Nil	Note[5]
Dilution by G.C., % wt. (D3525)	0.02	0.00	0.01	0.4	6.7[6]
Glycol Content (D2982)	Nil	Nil	Nil	Nil	Nil
Saponification No. (D94)	—	0.22	0.13	0.24	0.27
Carbon Distribution and Structural Group Analysis, n-d-m Method (D3238)					
% C_A	4.3	2.8	6.1	6.5	—
% C_N	29.6	29.7	30.4	40.7	—
% C_P	66.1	67.5	63.6	52.8	—
R_A	0.20	0.14	0.29	0.24	—
R_N	1.83	1.83	1.82	1.83	—
R_P	1.63	1.97	2.11	2.07	—
Total Chlorine, PPM (D1317)	—	—	<10[13]	<10[13]	—
Total Bromine, PPM	<16	—	<16	—	—
Metals PPM[7]					
Al, Ba, B, Ca, Cr, Cu, Fe, Pb, Mg, Ni, Si, Ag, Sn, Zn	<1[8]	<1[9]	<1[10]	<1[11]	<1[11]

Notes:
(1) Average of two samples.
(2) Average of three samples.
(3) Results determined by another laboratory.
(4) Average of two results (26 and 46).
(5) Unable to determine increasing values obtained during the test due to the high volatility of the oil.
(6) Due to relatively high volatility of the oil.
(7) For each metal.
(8) Average of two samples, determined by emission spectroscopy in another laboratory. <1 except for Ba and Si <5 in one sample.
(9) Determined by emission spectroscopy in another laboratory, <1 except for Al = 3, Ba and Si <5, Fe = 9.
(10) Average of three samples, determined by emission spectroscopy in another laboratory, <1 except for Ba and Si <5 in all samples, Fe and Pb <5 in one sample and Al, 10 in one sample.
(11) Determined by Atomic Absorption.
(12) Repeat results: 92.0, 7.1 and 0.9 respectively.
(13) Results obtained by Neutron Activation by a commercial laboratory were 0.2 ppm for "Z" and 0.4 ppm for the white oil.
(14) Basestocks X, Y and Z are solvent refined. All are used in formulating engine oils.

TABLE 13

RESPONSE TO ANTIOXIDANTS (ASTM D-943-54)(1)

OF

REFINED BASE STOCK OILS

TREATED WITH 0.3% 2,6-DITERTIARY-BUTYL PARA-CRESOL

BASE STOCK	X	Y	Z(2)		WHITE OIL	HYDRAULIC BASE
Test Duration			Total Acid Number D974, mg KOH/gm			
New Oil + Additive	0.01	0.03	0.01	0.02	0.02	0.02
168 hrs	0.04	0.00	0.00	0.04	0.00	0.06
336	0.05	0.00	0.00	0.08	0.02	0.05
504	0.05	0.13	0.03	0.04	0.00	0.05
672	0.09	0.03	0.00	0.05	0.00	0.06
840	0.16	0.00	0.03	0.04	0.00	0.05
1008	27.2	0.07	0.02	0.14	0.04	0.05
1176	–	–	0.02	–	0.02	0.00
1344	–	–	0.00	–	0.02	0.00
1512	–	–	0.00	–	0.05	0.02
1680	–	–	0.07	–	0.04	0.00
1848	–	–	0.28	–	0.07	0.02
2016	–	–	30.35	–	0.08	0.00
2184	–	–	–	–	–	0.00
2352	–	–	–	–	–	0.02
2520	–	–	–	–	–	0.02
2688	–	–	–	–	–	0.04
2856	–	–	–	–	–	0.09
3024	–	–	–	–	–	0.08

Notes: (1) 3 ml of sample drawn weekly for acidity test.
(2) Tests on samples of the oil received at different times.

TABLE 14

RESPONSE TO ANTIOXIDANTS (ASTM D-943-54)[1]

TO

REFINED BASE STOCK OILS

TREATED WITH 0.5% ZINC DIALKYLDITHIOPHOSPHATE

BASE STOCK	X	Y	Z[2]		WHITE OIL	HYDRAULIC BASE
Test Duration			Total Acid Number D974, mg KOH/gm			
New Oil + Additive	0.44	0.65	0.67	0.06	0.67	0.45
168 hrs	0.31	0.42	0.02	0.04	0.03	0.20
336	0.18	0.22	0.05	0.05	0.02	0.13
504	0.13	0.09	0.08	0.11	0.02	0.13
672	0.12	0.05	0.10	0.05	0.02	0.10
840	0.25	0.06	0.11	0.08	0.05	0.13
1008	0.22	0.05	0.15	0.11	0.03	0.23
1176	0.27	0.07	0.12	0.07	0.09	0.11
1344	0.23	0.11	0.12	0.10	0.07	0.09
1512	0.28	0.12	0.23	0.13	0.23	0.00
1680	13.73	0.11	0.18	0.18	0.10	0.00
1848	—	0.13	16.67	0.18	0.09	0.00
2016	—	0.07	—	0.19	0.20	0.03
2184	—	0.15	—	11.87	—	0.02
2352	—	18.12	—	—	—	0.00
2520	—	—	—	—	—	0.00
2688	—	—	—	—	—	0.09
2856	—	—	—	—	—	0.05
3024	—	—	—	—	—	0.04

Notes: (1) 3 ml of sample drawn weekly for acidity tests.
(2) Tests on samples of the oil received at different times.

TABLE 15

SIMULATED DISTILLATION DATA (MODIFIED D2887)(1)

OF

REFINED BASE STOCK OILS

REFINER OR TYPE OF BASE STOCK	X		Z(2)		WHITE OIL	HYDRAULIC BASE
Initial Boiling Point, °F	615	(686)	605	(671)	534	358
5% Distilled, °F	697	(709)	677	(692)	677	428
10% Distilled, °F	723	(739)	689	(707)	768	447
20% Distilled, °F	747	(743)	727	(725)	796	468
30% Distilled, °F	766	(754)	746	(736)	814	482
40% Distilled, °F	780	(760)	768	(739)	825	493
50% Distilled, °F	792	(769)	786	(757)	837	507
60% Distilled, °F	804	(781)	801	(771)	858	515
70% Distilled, °F	816	(789)	817	(782)	871	525
80% Distilled, °F	837	(802)	836	(808)	891	546
90% Distilled, °F	862	(828)	859	(840)	926	600
95% Distilled, °F	878	—	873	—	940	645
Final Boiling Point, °F	935	(905)	914	(916)	961	725

Notes: (1) Final column temperature 350°C. Vacuum distillation data in parenthesis; both "X" and "Z" showed 98% recovery.

(2) Average of three samples. Vacuum distillation data were obtained on a single sample.

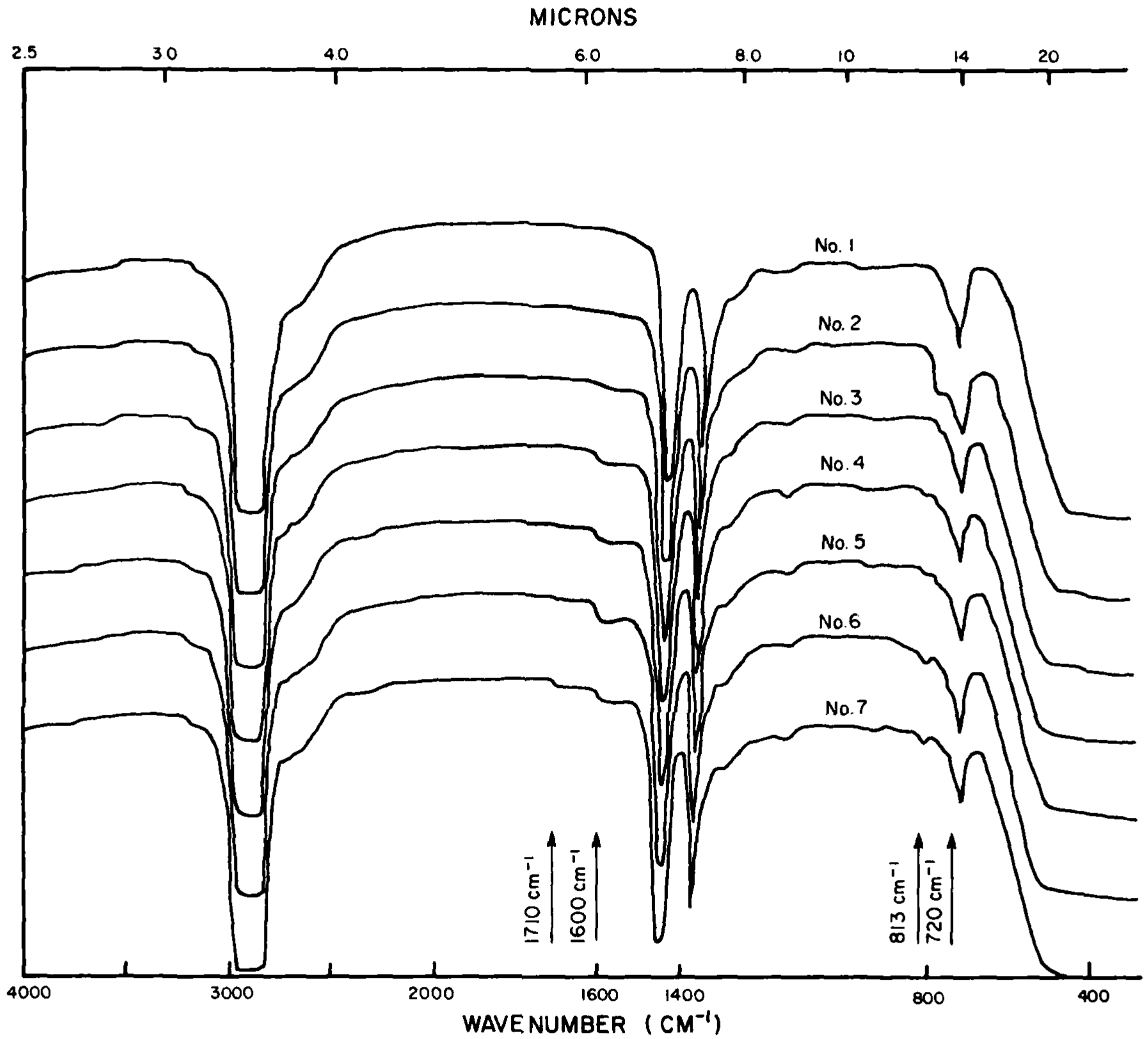

FIG. 1: IR SPECTRA OF SEVERAL BASESTOCKS

No. 1 White oil
No. 2 MIL-L-5606 basestock
No. 3 Virgin basestock
No. 4 Virgin basestock
No. 5 Canadian re-refined basestock
No. 6 Indian re-refined basestock
No. 7 RR re-refined basestock

APPENDIX A

DESCRIPTION OF ACID/CLAY RE-REFINING PROCESS

The used oil feedstock is initially flashed at about 150°C. The light oil/water overhead is separated and the oil fraction is retained for fuel. Following dehydration, the feedstock is treated with sulfuric acid for about 24-48 hours at about 40°C. This process coagulates oxidized oil contaminants and precipitates most of the metals contained in the feedstock. The oxidation products, metals, and acid form a sludge from which the oil is then separated. The treated oil is steam stripped and further refined by treatment with clay to remove most of the residual color bodies and colloidal carbon. The clarified oil is recovered from the oil/clay mixture by filtration (Fig. A1).

Typically, the amount of sulfuric acid (92-98% H_2SO_4) employed in the process varies from 4 percent to 7 percent by volume. One factor affecting acid consumption is the residual moisture content of the oil following dehydration. A higher residual moisture content requires that greater volumes of acid be used. The amount of clay used for clarification is about 0.5 pounds per Imperial gallon of acid treated oil.

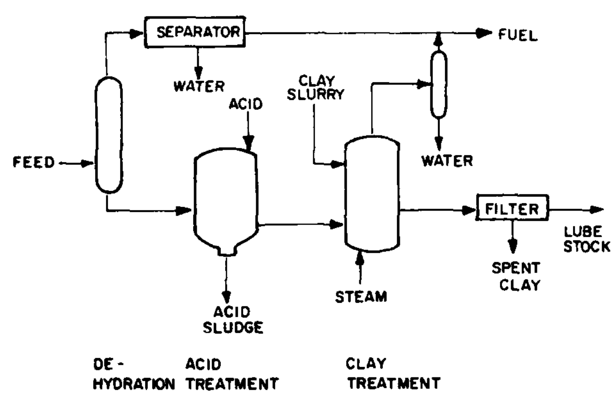

FIG. A1: RE-REFINING BY ACID/CLAY PROCESS

APPENDIX B

TABLE B1

PHYSICAL TEST DATA

OF ACID/CLAY PROCESS — REFINED BASE STOCKS (RE-REFINER D)

SAMPLE NO.	FLO-74402	FLO-74404(4)	FLO-74484	FLO-74485
DATE RECEIVED	74-10-08	74-10-08	74-10-08	74-10-08
ASTM Colour (D1500)	L4.0	D8.0	L3.5	3.5
API Gravity at 60°F (D287)	29.3	29.3	29.9	30.1
Specific Gravity at 60/60°F (D287)	0.8800	0.8800	0.8767	0.8756
Flash Point, °F (D92)	395	395	385	405
Copper Strip Corrosion (3 hrs. @ 212°F) (D130)	—	—	—	—
Viscosity at 100°F, cSt. (D445)	64.79	61.43	57.7	54.0
Viscosity at 210°F, cSt. (D445)	8.18	7.95	7.71	7.37
Viscosity Index (D2270)	103	104	106	106
Water and Sediment, % Vol. (D1796)	—	—	<0.001	0.004
Precipitation No. (D91)	—	—	<0.001	0.004
n-Pentane Insol., % wt. (D893/A)	0.08	0.11	0.02	—
Total Solids, mg/100 ml. (D2276)	—	—	19.3	6.3
Trace Sediment, % Vol. (D2273)	0.02	0.08	<0.005	—
AN-OIL-IZER	-3.0	-1.5	-6.5	-2.0
Appearance (Visual)	Clear(1)	Hazy(1)	Clear(1)	Clear(1)
Odour	Note (2)	Note (3)	Note (3)	Note (2)
Ramsbottom Carbon Residue, % wt. (D524)	0.14	0.32	0.10	—
Viscosity-Gravity Constant (D2501)	0.81	0.81	—	0.82
Molecular Weight (Est.) (D2502)	478	471	471	460
Refractive Index (20°C)	—	—	1.4831	1.4828

Notes: (1) FLO-74402 and FLO-74485 — clean, clear and free from visible impurities, FLO-74404 was hazy with some settled black sediment visible, FLO-74484 was clean, clear and free from visible impurities but showed a trace of black sediment on standing.

(2) Satisfactory — odour characteristic of re-refined base stocks produced by this process — slightly unpleasant — not like used oil.

(3) FLO-74404 definite sour odour, FLO-74484 — trace sour odour.

(4) Partially re-refined.

TABLE B2

CHEMICAL TEST DATA

OF ACID/CLAY PROCESS — RE-REFINED BASE STOCKS (RE-REFINER D)

SAMPLE NO.	FLO-74402	FLO-74404(6)	FLO-74484	FLO-74485
DATE RECEIVED	74-10-08	74-10-08	74-10-08	74-10-08
Sulphated Residue, % wt. (D874)	0.000	0.176	0.000	0.002
Aniline Point, °F (D611)	225.3	221.1	223.0	—
Total Acid No. (D664)	0.20	0.28	—	—
Total Base No. (D664)	0.00	0.00	—	—
Hydrocarbon Types (D2007)				
Saturates, % wt.	76.7	72.8	75.5	77.1
Aromatics, % wt.	21.1	22.1	22.7	21.4
Polar Compounds, % wt.	2.2	5.1	1.8	1.4
Dilution by GC, % wt. (D3525)	—	—	0.02	—
Glycol Content (D2982)	—	—	Nil	—
Saponification No. (D94)	1.40	1.01	—	—
Metals, ppm (by Emission Spectroscopy)(1)				
Al, B, Cr, Cu, Fe, Pb, Mg, Ni, Ag, Si, Zn, Sn	<1(2)	<1(3)	<1(4)	<1(5)

Notes: (1) Determined by a commercial laboratory.
(2) Except for Al — 6, Fe < 5, Pb < 5, Si — 10.
(3) Except for Al — 8, Fe — 23, Pb — 350, Si — 16, Zn < 1000.
(4) Except for Fe < 10, Pb < 10, Zn — undetermined.
(5) Except for Al — 2, Fe < 5, Pb < 5, Mg — 6, Si — 8.
(6) Partially re-refined.

APPENDIX C

TABLE C1

PHYSICAL TEST DATA

OF ACID/CLAY PROCESS — RE-REFINED BASE STOCKS (RE-REFINER E)

SAMPLE NO. DATE RECEIVED	NRL-26857 67-02-10	NRL-26957 67-04-05	NRL-27087 67-06-26	NRL-28085 67-08-29	NRL-28089 67-09-06	NRL-28116 67-09-18
ASTM Colour (D1500)	5.0	L6.5	4.5	L4.0	L3.5	L5.5
API Gravity at 60°F (D287)	28.8	28.9	28.9	29.1	29.5	29.2
Specific Gravity at 60/60°F (D287)	0.8827	0.8822	0.8822	0.8811	0.8789	0.8805
Flash Point, °F (D92)	460	455	460	455	—	440
Copper Strip Corrosion (3 hrs @ 212°F) (D130)	No. 1	—	—	No. 1	—	No. 1
Cloud Point, °F (D2500)	+16	+14	+18	+15	—	+19
Pour Point, °F (D97)	-25	-25	-25	-25	—	-20
Viscosity at 100°F, cSt. (D445)	73.27	75.80	80.97	74.26	69.40	73.16
Viscosity at 210°F, cSt. (D445)	8.90	9.09	9.42	8.94	8.58	8.92
Viscosity Index (D2270)	104	103	101	103	104	104
Trace Sediment, % Vol. (D2273)	0.004	—	—	<0.001	<0.001	0.000
Appearance (Visual)	Note (1)	Note (1)	Note (1)	Note (1)	Note (1)	Note (1)
Odour	Note (2)	Note (2)	Note (2)	Note (2)	Note (2)	Note (2)
Ramsbottom Carbon Res., % wt. (D524)	0.12	0.24	0.17	0.18	—	0.17
Viscosity-Gravity Const., (D2501)	0.84	0.84	0.84	0.84	0.84	0.84
Molecular Weight (Est.) (D2502)	481	488	496	485	479	483
Foaming (D892)						
Tendency at 75°F, ml.	450	600	560	560	—	550
Tendency at 200°F, ml.	25	70	60	80	—	110
Tendency at 75°F, after 200°F, ml.	290	520	530	550	—	650
Stability at all cond., ml. (after 10 min.)	205-0-5	230-5-190	165-0-110	70-0-50	—	160-0-80

Notes: (1) Clean, clear and free from visual impurities.
(2) Satisfactory — odour characteristic of re-refined base stocks produced by this process — slightly unpleasant — not like used oil.

TABLE C2

CHEMICAL TEST DATA

OF ACID/CLAY PROCESS — RE-REFINED BASE STOCKS (RE-REFINER E)

SAMPLE NO. DATE RECEIVED	NRL-26857 67-02-10	NRL-26957 67-04-05	NRL-27087 67-06-26	NRL-28085 67-08-29	NRL-28089 67-09-06	NRL-28116 67-09-18
Ash, % wt. (D482)	0.005	—	—	0.03	—	0.02
Total Sulphur, % wt. (D129)	0.15	—	0.22	0.09	—	0.15
Aniline Point, °F (D611)	225.0	225.7	227.5	226.0	225.4	225.0
Glycol Content (D2982)	—	—	Nil	—	—	Nil
Strong Acid No. (D664)	Nil	Nil	Nil	Nil	—	Nil
Total Acid No. (D664)	0.12	0.37	0.13	0.30	—	0.24
Total Base No. (D664)	0.00	0.00	0.00	0.00	—	0.01
Metals, ppm(1)						
Ca	200	—	—	600	—	300
Ba	400	—	—	—	—	300
Zn	300	—	—	0	—	100

Note: (1) Determined by proposed ASTM complexometric method PG. 1016, Part 17, 1965.

APPENDIX D

TABLE D1

PHYSICAL TEST DATA

OF ACID/CLAY PROCESS — RE-REFINED BASE STOCKS (RE-REFINER F)

SAMPLE NO.	NRL-28673	NRL-28720	NRL-28836	FLO-74335	FLO-75090	FLO-76381	FLO-78160	AVG.	"S"(5)
DATE RECEIVED	68-10-2	68-11-20	69-02-12	74-07-19	75-04-08	76-12-29	78-05-04		
ASTM Colour (D1500)	L2.0	3.0	2.5	L3.0	3.5	3.5	2.5	—	—
API Gravity at 60°F (D287)	29.9	29.1	29.8	29.6	29.8	29.1	29.7	29.6	0.34
Specific Gravity at 60/60°F (D287)	0.8767	0.8811	0.8772	0.8783	0.8772	0.8811	0.8778	0.8785	0.0019
Flash Point, °F (D92)	420	440	430	415	410	420	390	418	16
Copper Strip Corr. (3 hrs. @ 212°F) (D130)	No. 1a	No. 1a	No. 1a	No. 2b	No. 1a	No. 1a	No. 1a	—	—
Cloud Point, °F (D2500)	+16	+18	+18	+20	—	+24(1)	+18	+19	—
Pour Point, °F (D97)	-15	0	-10	+ 5	—	- 5	+10	- 2.5	9
Viscosity at 100°F, cSt. (D445)	54.76	67.48	59.70	58.43	—	65.67	54.11	60.03	5.53
Viscosity at 210°F, cSt. (D445)	7.26	8.23	7.74	7.63	7.33	8.05	7.26	7.64	0.39
Viscosity Index (D2270)	100	98.5	102	102	—	97	102	100	2.1
Precipitation No. (D91)	—	—	—	—	—	0.000	0.000	0.00	—
n-Pentane Insol., % wt. (D893/A)	—	—	—	0.04	—	0.01	0.00	0.02	0.02
Total Solids, mg/100 ml. (D2276)	—	—	—	1.68	2.72	5.3	3.5	3.30	1.53
Trace Sediment, % Vol. (D2273)	0.000	0.001	0.001	0.001	0.001	0.000	0.001	0.001	0.000
An-Oil-Izer	—	—	—	- 2.0	- 4.0	- 4.0	- 2.0	- 3	1.2
Appearance (Visual)	Clear(2)	Clear(2)	Clear(2)	—	—	Clear	Clear(3)	—	—
Odour	Note (4)	Note (4)	Note (4)	Note (4)	Note (4)	Note (4)	Note (4)	—	—
Ramsbottom Carbon Residue, % wt. (D524)	0.11	0.17	0.14	0.16	—	0.16	0.12	0.14	0.02
Viscosity-Gravity Constant (D2501)	0.82	0.82	0.82	0.82	—	0.82	0.85	0.83	0.01
Molecular Weight (Est.) (D2502)	449	465	466	459	—	475	448	460	10.5
Foaming (D892)									
Tendency at 75°F, ml	430	510	530	—	470	200	430	428	119
Tendency at 200°F, ml	30	40	45	—	50	30	35	38	8
Tendency at 75°F, after 200°F, ml	420	450	480	—	660	200	400	435	148
Stability at all Cond., ml (after 10 min.)	10-0-10	0	10-0-5	—	20-0-40	0	0		
Emulsion Characteristics (D1401)	—	—	—	—	40-40-0(15)	40-39-1(35)	40-31-9(60)		

Notes:
(1) Approximate cloud point; entire sample tended to get cloudy at one time, possibly due to the presence of trace additive.
(2) Clean, clear and free from visible impurities.
(3) Excellent — resembles a virgin oil, clean, crystal clear and free from visible impurities.
(4) Satisfactory — odour characteristic of re-refined base stocks produced by this process — slightly unpleasant — *not* like used oil.
(5) "S" is standard deviation.

TABLE D2

CHEMICAL TEST DATA(1)

OF ACID/CLAY PROCESS — RE-REFINED BASE STOCKS (RE-REFINER F)

SAMPLE NO. / DATE RECEIVED	NRL-28673 68-10-2	NRL-28720 68-11-20	NRL-28836 69-02-12	FLO-74335 74-07-19	FLO-75090 75-04-08	FLO-76381 76-12-29	FLO-78160 78-05-04	AVG.	"S"(6)
Ash, % wt. (D482)	0.00	0.00	0.00	0.000	0.000	0.000	0.000	0.00	0
Total Sulphur, % wt. (D129)	0.08	0.17	0.10	0.16	0.09	0.13	0.21	0.13	0.05
Nitrogen, ng/μL (Microcoulometer, Dohrmann)	—	—	—	—	23	33	5	20	—
Aniline Point, °F (D611)	222.6	223.2	224.8	220.0	—	225.9	221.8	223.1	2.1
Hydrocarbon Types (D2007)									
Saturates, % wt.	—	—	—	76.7	75.9	78.0	77.9	77.1	1.0
Aromatics, % wt.	—	—	—	21.1	22.2	20.4	20.3	21.0	0.9
Polar Compounds, % wt.	—	—	—	2.2	1.9	1.6	1.9	1.9	0.2
Strong Acid No. (D664)	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Total Acid No. (D664)	0.04	0.10	0.06	0.20	0.11	0.11	0.12	0.11	0.05
Total Base No. (D664)	0.00	0.00	0.00	0.09	0.00	0.01	0.00	0.01	0.03
IpH Reading (D664)	—	—	—	7.9	6.0	8.3	6.0	7.1	1.2
Crankcase Dilution, % Vol. (D322)	—	—	—	—	—	Nil	Nil	Nil	—
Dilution by GC, % wt. (D3525)	—	—	—	0.03	<0.01	0.05	0.05	0.03	0.02
Glycol Content (D2982)	Nil	Nil	Nil	—	Nil	Nil	Nil	Nil	—
Saponification No. (D94)	—	—	—	1.75	0.5	0.12	0.28	0.66	0.7
Metals, ppm (AA)(2)									
Cu, Fe, Pb, Al, Cr, Mg, Ca, Ba, Zn, Si	<100(3)	<100(3)	<100(3)	—	<1(4)	<1(5)	<1	—	—
Carbon, % wt. (240 anal.)	—	—	—	—	—	—	86.6	—	—
Hydrogen, % wt. (240 anal.)	—	—	—	—	—	—	13.5	—	—

Notes:
(1) Nitrogen, carbon and hydrogen analyses were performed by Dept. of Energy, Mines and Resources.
(2) For each metal.
(3) Ca, Ba, Zn only — determined by proposed ASTM complexometric method, pg. 1016, Part 17, 1965.
(4) Except for Al — 2 ppm, Ba — <5 ppm, Cr — 3 ppm, Fe — 6 ppm, Sn — 2 ppm.
(5) Except for Si — 2 ppm.
(6) "S" is standard deviation.

TABLE D3

RESPONSE TO ANTIOXIDANTS (ASTM D943-54)[1]

TREATED WITH 0.3% 2,6-DITERTIARY-BUTYL PARA-CRESOL (BASE STOCK — RE-REFINER F)

SAMPLE NO. DATE RECEIVED		FLO-75090 75-04-08	FLO-76381 76-12-29	FLO-78160[2] 78-05-04	
Test Duration	*Total Acid No. D974, mg KOH/gm*				
New Oil and Additive		0.09	0.09	0.09	0.09
168 hours		1.45	0.15	0.11	0.15
336 hours		0.32	1.04	0.21	0.14
504 hours		11.02	22.00	0.41	0.51
672 hours		—	—	15.75	15.51
840 hours		—	—	—	—
1008 hours		—	—	—	—

Notes: (1) 3 ml of sample drawn weekly.
(2) Duplicate Determinations.

TABLE D4

RESPONSE TO ANTIOXIDANTS (ASTM D943-54)[1]

TREATED WITH 0.5% ZINC DIALKYLDITHIOPHOSPHATE (BASE STOCK — RE-REFINER F)

SAMPLE NO.		FLO-75090	FLO-76381	FLO-78160[2]	
DATE RECEIVED		75-04-08	76-12-29	78-05-04	
Test Duration	Total Acid No. D974, mg KOH/gm				
New Oil and Additive		0.56	0.81	0.67	0.49
168 hours		0.31	0.39	0.29	0.18
336 hours		0.19	0.21	0.29	0.15
504 hours		0.21	0.42	0.33	0.17
672 hours		0.33	0.54	0.30	0.22
840 hours		0.49	16.57	0.44	0.45
1008 hours		18.22	—	0.63	9.91
1176 hours		—	—	11.46	—

Notes: (1) 3 ml of sample drawn weekly.
(2) Duplicate Determinations.

TABLE D5

SIMULATED DISTILLATION DATA (MODIFIED D2887) (BASE STOCK — RE-REFINER F)(1)

SAMPLE NO. DATE RECEIVED	NRL-28673 68-10-02	NRL-28720 68-11-20	NRL-28836 69-02-12	FLO-74335 74-07-19	FLO-75090 75-04-08	FLO-76381 76-12-29	FLO-78160 78-05-04	AVG.	"S"(3)
Initial Boiling Point, °F	—	—	—	563	563 (509)	615 (588)	591	583	25
5% Distilled, °F	—	—	—	656	664 (660)	696 (704)	658	669	19
10% Distilled, °F	—	—	—	686	698 (701)	723 (720)	692	700	16
20% Distilled, °F	—	—	—	715	730 (730)	752 (752)	723	730	16
30% Distilled, °F	—	—	—	738	752 —	776 (775)	749	754	16
40% Distilled, °F	—	—	—	762	772 —	797 (797)	773	776	15
50% Distilled, °F	—	—	—	778	790 (805)	809 (813)	788	791	13
60% Distilled, °F	—	—	—	797	807 (847)	823 (851)	813	810	11
70% Distilled, °F	—	—	—	817	820 —	834 (886)	836	827	10
80% Distilled, °F	—	—	—	835	839 (936)	855 (925)	855	846	13
90% Distilled, °F	—	—	—	879	861 (1005)	876 (985)	904	880	18
95% Distilled, °F	—	—	—	895	874 —	894 —	926	897	22
96% Distilled, °F	—	—	—	898	878 —	— —	—	888	10
Final Boiling Point, °F(2)	—	—	—	911	913 (1023)	926 (1083)	945	924	16

Notes: (1) Final column temperature, 350°C. *Figures in parentheses were obtained by vacuum distillation*; recovery 93% for FLO-75090 and 93% for FLO-76381.
(2) Tailings at the end; the final gc boiling points should be higher. See vacuum distillation data in parentheses.
(3) "S" is standard deviation.

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As shown, when well re-refined, the base stocks have excellent properties including a good response to anti-oxidants and a high degree of cleanliness.

UNCLASSIFIED

1. Lubricating oils — reclamation.
2. Waste oil.
3. Acid/clay re-refining.

I. Strigner, P.L.
II. Moroz, G.
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